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IS 557 (1988): Sodium acetate, photographic grade [CHD 5: Electroplating Chemicals and Photographic Materials]



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Indian Standard

**SPECIFICATION FOR SODIUM ACETATE,
PHOTOGRAPHIC GRADE**

(Third Revision)

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BUREAU OF INDIAN STANDARDS

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SODIUM ACETATE, PHOTOGRAPHIC GRADE

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards on 1 January 1988, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1954 and subsequently revised in 1968 and 1978. This standard earlier contained technical as well as photographic grade of the material. In the present revision, only photographic grade of the material has been retained, since the Committee responsible for the formulation of this standard felt that the technical grade of the material should be covered under a separate standard. The limit for anhydrous sodium acetate content has also been modified in this revision.

0.3 This Indian Standard is one of a series of specifications for photographic grade chemicals which are commonly used in the processing of sensitized

photographic materials. These specifications have been prepared to establish criteria of purity which will provide a practical and economical grade, and prevent possible faulty processing which might be caused by chemicals of inferior quality, and to furnish manufacturers, suppliers and processors with reliable and readily available specifications for photographic chemicals of satisfactory quality.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for sodium acetate (hydrated) ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) and sodium acetate (anhydrous) (CH_3COONa) used in the processing of photographic materials.

2. GRADES

2.1 There shall be the following two grades of the material:

- a) Hydrated, and
- b) Anhydrous.

3. REQUIREMENTS

3.1 Description

3.1.1 Sodium Acetate, Hydrated — shall be in the form of snow white crystals, free from extraneous impurities.

3.1.2 Sodium Acetate, Anhydrous — shall be in the form of snow white powder free from extraneous impurities.

3.2 The material shall comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 6 of Table 1.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable air-tight containers.

4.2 Marking — The packages shall be securely closed and marked legibly and indelibly with the following information:

- a) Name and grade of the material;
- b) Mass of the material in the package;
- c) Name of the manufacturer and his recognized trade-mark, if any; and
- d) Lot or batch number, in code or otherwise.

4.2.1 The packages may also be marked with the Standard mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

TABLE 1 REQUIREMENTS FOR SODIUM ACETATE, PHOTOGRAPHIC GRADE
(Clause 3.2)

Sl. No.	CHARACTERISTIC	REQUIREMENTS FOR GRADE		METHOD OF TEST (REF TO CL No. IN APPENDIX A)
		Hydrated (3)	Anhydrous (4)	
(1)	(2)			(5)
i)	Sodium acetate content:			
a)	Hydrated (calculated as $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$), percent by mass, <i>Min</i>	98.5	—	A-2
b)	Anhydrous (calculated as CH_3COONa), percent by mass, <i>Min</i>	—	98.8	A-2
ii)	Insoluble matter (as calcium, magnesium and ammonium hydroxide precipitate), percent by mass, <i>Max</i>	0.3	0.5	A-3
iii)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.003	0.005	A-4
iv)	Iron (as Fe), percent by mass, <i>Max</i>	0.003	0.005	A-5
v)	Free alkali (as NaOH), percent by mass, <i>Max</i>	0.02	0.04	A-6
vi)	Free acid (as CH_3COOH), percent by mass, <i>Max</i>	0.06	0.1	A-6
vii)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.2	0.2	A-7
viii)	Clarity of 10 percent solution	Clear and colourless	Clear and colourless	A-8

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in Appendix B.

APPENDIX A

(Clause 3.2)

METHODS OF TEST FOR SODIUM ACETATE, PHOTOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS:1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF SODIUM ACETATE

A-2.1 Reagents

A-2.1.1 *Standard Sulphuric Acid* — 0.5 N.

A-2.1.2 *Blue Litmus Paper* — Strips of 0.6 × 5 cm.

A-2.1.3 *Methyl Orange Indicator* — Dissolve 0.01 g of methyl orange in 100 ml of water.

A-2.1.4 *Standard Sodium Hydroxide Solution* — 0.5 N.

A-2.2 **Procedure** — Weigh accurately about 0.5 g of the material and ignite it in a platinum crucible protected from flame, gently at first then at low red heat until thoroughly carbonized. Cool the crucible and place it in a beaker. At 75 ml of water and 50 ml of standard sulphuric acid and make sure that the crucible is covered completely by the liquid. Cover the beaker with a watch glass

and boil gently for about 30 minutes. Filter the contents of the beaker and wash the beaker, the watch glass and the filter paper thoroughly with hot water until the washings cease to turn blue litmus paper red. Cool the filtrate to room temperature, add 2 drops of methyl orange indicator and titrate the excess of acid with standard sodium hydroxide solution.

A-2.2.1 Run a blank in the same manner with the same quantities of all the reagents but without using the material under test.

A-2.3 Calculation

a) Sodium acetate (as $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$), percent by mass = $\frac{13.609 (V_1 - V_2) N}{M}$

b) Sodium acetate (as CH_3COONa), percent by mass = $\frac{8.204 (V_1 - V_2) N}{M}$

where

V_1 = volume in ml of standard sodium hydroxide solution required for the blank,

V_2 = volume in ml of standard sodium hydroxide solution required for the test with material,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

*Specification for water for general laboratory use (second revision).

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Reagents

A-3.1.1 Ammonium Oxalate Solution — 4 percent.

A-3.1.2 Ammonium Phosphate Solution — 10 percent.

A-3.1.3 Ammonium Hydroxide Solution — 1 : 9.

A-3.1.4 Dilute Ammonium Hydroxide Solution — 1 : 39.

A-3.2 Procedure — Dissolve 10 ± 0.1 g of the material in 75 ml of water. Add 10 ml of ammonium oxalate solution, 4 ml of ammonium phosphate solution and 20 ml of dilute ammonium hydroxide. Allow to stand overnight. If any precipitate is formed, filter and wash with dilute ammonium hydroxide. Dry and ignite at dull red heat (approximately 600°C). Cool in a desiccator and weigh.

A-3.3 Calculation

Insoluble matter (as calcium, magnesium and ammonium hydroxide precipitate), percent by

$$\text{mass} = \frac{100 \times M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-4. TEST FOR HEAVY METALS

A-4.1 Apparatus

A-4.1.1 Nessler Cylinders — 50 ml capacity.

A-4.2 Reagents

A-4.2.1 Standard Lead Solution — Dissolve 1.60 g of lead nitrate [$\text{Pb}(\text{NO}_3)_2$] in water and make up the volume to 1 000 ml. Dilute 10 ml of this solution with water and make up the volume to 1 000 ml. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-4.2.2 Dilute Hydrochloric Acid — approximately 1 N.

A-4.2.3 Hydrogen Sulphide Solution — Prepare a fresh, saturated aqueous solution of hydrogen sulphide gas.

A-4.3 Procedure — Dissolve 5.000 g of the material in water and make up to 50 ml with water. Take 10 ml of the solution into a Nessler cylinder and add one of the following quantities of standard lead solutions, depending on the grade of sodium acetate:

- 6 ml of standard lead solution in case of hydrated grade, and
- 10 ml of standard lead solution in case of anhydrous grade.

Add 1 ml of dilute hydrochloric acid, 10 ml of hydrogen sulphide solution and make up the volume to 50-ml mark. To the other cylinder add 30 ml of the remaining portion of the solution, 1 ml

of dilute hydrochloric acid, 10 ml of hydrogen sulphide solution and make up the volume with water to 50-ml mark.

A-4.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the second cylinder is not darker than that produced in the first.

A-5. TEST FOR IRON

A-5.1 Apparatus

A-5.1.1 Nessler Cylinders — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 Butanolic Potassium Thiocyanate Solution — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-5.2.2 Ammonium Persulphate — solid.

A-5.2.3 Concentrated Hydrochloric Acid — See IS : 265-1976*.

A-5.2.4 Standard Iron Solution — Dissolve 0.8635 g of ferric ammonium sulphate [$\text{Fe}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid (10 percent by volume) and dilute with water to 1 000 ml. Dilute 10 ml of this solution to 100 ml with water. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-5.3 Procedure — Dissolve 1.000 g of the material in 10 ml of water. Add 1 ml of hydrochloric acid, about 30 mg of solid ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution.

A-5.3.1 Carry out a control test using the same quantities of all the reagents except the material and the appropriate quantity of standard iron solution prescribed below, depending on the grade of sodium acetate:

- 3 ml of standard iron solution in case of hydrated grade, and
- 5 ml of standard iron solution in case of anhydrous grade.

Make up the volume of the test and control solutions to 50 ml. Shake the solutions vigorously for 30 seconds, allow the layers to separate and then compare the colour of the butanol layers after five minutes.

A-5.3.2 The limits prescribed in Table 1 shall be taken as not having been exceeded if any red colour produced in the butanol layer of the first cylinder, is not darker than that produced in the second cylinder.

A-6. DETERMINATION OF FREE ALKALI AND FREE ACID

A-6.1 Reagents

A-6.1.1 Phenolphthalein Indicator — Dissolve 0.1 g of phenolphthalein in 100 ml of rectified spirit

*Specification for hydrochloric acid (second revision).

(see IS : 323-1959*) which has been previously neutralized to the indicator.

A-6.1.2 Standard Hydrochloric Acid — 0.02 N.

A-6.1.3 Standard Sodium Hydroxide Solution — 0.02 N.

A-6.2 Procedure for Free Alkali — Weigh accurately about 5 g of the material and dissolve in 50 ml of freshly distilled water. Add 3 drops of phenolphthalein indicator. If pink colour is not produced, the material is free from alkali. If pink colour is produced, quickly titrate with standard hydrochloric acid.

A-6.3 Procedure for Free Acid — If pink colour is not produced on adding phenolphthalein indicator to the solution of the material as described under A-6.2, quickly titrate with standard sodium hydroxide solution.

A-6.4 Calculation — Calculate the percentage of free alkali as sodium hydroxide (NaOH) as given under A-6.4.1, and of free acid as given under A-6.4.2.

A-6.4.1 Free alkali (as NaOH), percent by mass

$$= \frac{4.0 V N}{M}$$

where

V = volume in ml of standard hydrochloric acid required for the titration,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for the test.

A-6.4.2 Free acid (as CH_3COOH), percent by

$$\text{mass} = \frac{6.0 V N}{M}$$

where

V = volume in ml of standard sodium hydroxide solution required for the titration,

N = normality of standard sodium hydroxide solution, and

*Specification for rectified spirit (revised).

M = mass in g of the material taken for the test.

A-7. TEST FOR CHLORIDES

A-7.1 Apparatus

A-7.1.1 Nessler Cylinders — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 Dilute Nitric Acid — approximately 4 N.

A-7.2.2 Silver Nitrate Solution — approximately 5 percent (m/v).

A-7.2.3 Standard Sodium Chloride Solution — Dissolve 0.1649 g of sodium chloride in water and make up the volume to 1000 ml. Pipette 100 ml of this solution and dilute it to 1000 ml. One millilitre of this solution contains 0.01 mg of chloride (as Cl).

A-7.3 Procedure — Dissolve 2.000 g of the material in water and make up the volume to 500 ml. Transfer 5 ml of the solution into a Nessler cylinder. Add 1 ml of dilute nitric acid, 1 ml of silver nitrate solution and make up the volume to 50 ml. Carry out a control test in the other Nessler cylinder using 6 ml of standard sodium chloride solution and the same quantities of other reagents and finally diluting to 50 ml. Stir the solutions immediately with a glass rod and set aside for five minutes. Compare the opalescence produced in the two cylinders.

A-7.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced in the test with the material is not greater than that produced in the control test.

A-8. CLARITY OF 10 PERCENT SOLUTION

A-8.1 Procedure — Dissolve 10 g of the material in 100 ml of water and transfer to a 100-ml measuring cylinder. In another measuring cylinder, take 100 ml of water. View the solution from above against black and white background. The solution shall show not more than a slight flocculence after 24 hours.

NOTE — The solution of the material should be prepared fresh.

APPENDIX B

(Clause 5.1)

SAMPLING OF SODIUM ACETATE, PHOTOGRAPHIC GRADE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling

instrument and the containers for samples from adventitious contamination.

B-1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.3 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.4 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of this standard.

B-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

LOT SIZE N (1)	NUMBER OF CONTAINERS TO BE SELECTED n (2)
Up to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

LOT SIZE N (1)	NUMBER OF CONTAINERS TO BE SELECTED n (2)
Up to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-2.3 These containers shall be selected at random. In order to ensure randomness of selection, random sampling procedures given in IS : 4905-1968* may be followed. Alternatively, the following procedure may be adopted:

Starting from any container, count them 1, 2, 3,....., up to r and so on in one order, where r is the integral part of N/n , N being the lot size and n the sample size. Every r th container thus counted shall be taken out for drawing samples.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under 3 and shall not exceed 1 kg.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of

these portions; a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under B-1.4. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (see B-3.1.2) and a set of individual samples (see B-3.1.3) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Test for sodium acetate content shall be carried out on each individual sample.

B-4.2 Tests for all the remaining characteristics mentioned in Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Sodium Acetate Content — All the test results for sodium acetate shall be recorded. The mean and the range of these test results shall be calculated as follows:

Mean (\bar{X}) = sum of the test results divided by the number of test results, and

Range (R) = difference between the maximum and the minimum values of the test results.

The lot shall be declared to have satisfied the requirement for sodium acetate content if the value of the expression ($\bar{X} - 0.6 R$) is not less than the specified limit.

B-5.2 For Composite Sample — The test results on the composite sample shall meet the corresponding requirements specified in Table 1 for the relevant grade.

B-5.3 The lot shall be declared as conforming to the requirements of this standard if the different test results obtained under B-5.1 and B-5.2 meet the corresponding requirements given in Table 1.

*Methods for random sampling.

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